IS 4359 (1967): Kattha [FAD 8: Food Additives]

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IS : 4359 - 1967
(Reaffirmed 1992)

Indian Standard

SPECIFICATION FOR *KATTHA*

(Second Reprint OCTOBER 1997)

UDC 633.875.28

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 6 *December* 1967

AMENDMENT NO. 1 MAY 1994
TO
IS 4359 : 1967 SPECIFICATION FOR *KATTHA*

(*Page* 5, *clause* **3.1.2**):

(a) Add the following sentence after the first sentence:

'The material shall also be free from titanium dioxide.'

(b) Add the following clause after **3.1.2**:

3.1.3 The material shall be free from gambier *KATTHA* when tested by the following method:

'Take of 0.3 g of the material and warm with 2 ml of 90 percent alcohol, cool and filter. Add 2 ml of a solution of sodium hydroxide, shake and allow to separate. Absence of a brilliant green fluorescence in the upper layer indicates absence of gambier *KATTHA*.'

(FAD 8)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 DECEMBER 1978

TO

IS:4359-1967 SPECIFICATION FOR *KATTHA*

Alteration

[*Page 6, Table 1, col 3, 4 and 5, against Sl No.* (i)] - Substitute '16' *for* '12' at all the places.

Addendum

(*Page 5, clause 3.2*) - Add the following new clause after 3.2:

'3.2.1 In case the moisture content is less or more than 16 percent (*on as received basis*), weight of the consignment shall be adjusted on the basis of 16 percent moisture.'

(CDC 36)

Printed at Simco Printing Press, Delhi, India

AMENDMENT NO. 2 MAY 1994
TO
IS 4359 : 1967 SPECIFICATION FOR *KATTHA*

(*Page* 5, *clause* 3.1.2):

(a) Add the following sentence after the first sentence:

'The material shall also be free from titanium dioxide.'

(b) Add the following clause after 3.1.2:

3.1.3 The material shall be free from gambier *KATTHA* when tested by the following method:

'Take of 0.3 g of the material and warm with 2 ml of 90 percent alcohol, cool and filter. Add 2 ml of a solution of sodium hydroxide, shake and allow to separate. Absence of a brilliant green fluorescence in the upper layer indicates absence of gambier *KATTHA*.'

(FAD 8)

Printed at Simco Printing Press, Delhi, India

Indian Standard

SPECIFICATION FOR *KATTHA*

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(*Continued on page* 2)

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard
SPECIFICATION FOR *KATTHA*

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 October 1967, after the draft finalized by the *KATTHA*, Vegetable Tans and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 *KATTHA* (कत्था) is obtained mostly by crystallization in cold from the water extractives of the heartwood of *Acacia catechu* Willd., fam. Leguminosae commonly known as *KHAIR* (खैर) tree, which is widely distributed in India, from the north-west plains to eastwards in Assam and throughout the country, particularly in the deciduous and drier regions. The other species namely, *A. chundra* Willd and *A. catechuoides* Benth are sometimes used for extraction of *KATTHA*. The manufacture of *KATTHA* is carried out both, as a cottage scale industry in and around the forests abounding in *KHAIR* trees, and also in the organized sector. The general principle of extraction of the heartwood and of consequent separation of *KATTHA* (containing most of catechin) from cutch (principally catechu-tannic acid) is the same in cottage sector as well as large scale units. In the conventional process of *KATTHA* manufacture followed by cottage sector of the industry, during separation of the crystalline *KATTHA*, the soluble cutch-tans, a valuable byproduct are neither effectively removed nor recovered, except by a few.

0.3 *KATTHA* is one of the principal ingredients used in the preparation of *PAN* (पान) from betel leaves, for chewing purposes when, in combination with lime, it gives the characteristic red colouration. In Ayurvedic and Unani systems of medicine, *KATTHA* is used as astringent; cooling and digestive; useful in relaxed conditions of throat, mouth and gums; and also in cough and diarrhoea. Externally, it is employed as an astringent and as a cooling application to ulcers, boils and eruptions of the skin.

0.4 A rational specification for *KATTHA*, fair both to manufacturers as well as consumers, should take into account its main constituent, namely, the catechin. Unfortunately, the specifications laid down so far in the Prevention of Food Adulteration (PFA) Rules, 1955, Ministry of Health and Family Planning, Government of India have overlooked this very important aspect. This has given rise to anomalous position. Consequently the

Central Committee for Food Standards (CCFS) requested ISI to undertake the task of evolving rational standards for *KATTHA*. An Indian Standard specification (IS : 2962-1964*) formulated as a result of collaborative testing over a number of years paved the foundation for testing *KATTHA* and made the task of preparation of specification a reality. It is hoped that this specification will be adopted shortly by the CCFS for the purposes of the PFA Rules, 1955.

0.5 This specification has been formulated on the basis of collaborative tests carried out on a large number of samples of *KATTHA*, obtained from the cottage sector as well as large scale manufacturers, in the laboratories of Central Food Technological Research Institute (CSIR), Mysore; Italab Private Ltd, Bombay; The Public Analyst, Government of Uttar Pradesh, Lucknow; Central Drugs Laboratory, Calcutta; and the Indian Standards Institution. The co-operation received from them is gratefully acknowledged. The limits for poisonous metals are the same as specified in the PFA Rules, 1955, Ministry of Health & Family Planning, Government of India.

0.5.1 Assistance has also been derived from the following publications:

SADGOPAL. Development of *KATTHA* and cutch industry. *Research and Industry*. 3, 7; 1958; 186 - 91.

Wealth of India — Industrial Products. Part V. 1964. Council of Scientific and Industrial Research, New Delhi. P 149 - 54.

0.6 This standard contains clause **4.1** which permits the purchaser to use his option for selection to suit his requirements.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for *KATTHA* (कत्था) obtained from the heartwood extractives of *Acacia catechu* Willd., *A. chundra* and *A. catechuoides* Benth, fam. Leguminosae. The material is generally used in the preparation of *PAN* (पान) for chewing, and sometimes for pharmaceutical uses in Ayurvedic and Unani systems of medicine.

*Methods of sampling and test for *KATTHA*.

†Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 There shall be three grades of the material, namely, Grade 1, Grade 2 and Grade 3 (*see* Table 1).

3. REQUIREMENTS

3.1 Description — The material shall be obtained by suitably crystallizing out the catechin-rich fraction from the concentrated and cooled extractives of the heartwood of *Acacia catechu* Willd., *A. chundra* Willd., and *A. catechuoides* Benth fam. Leguminosae. It shall be comparatively free from the water-soluble catechu-tannic acid or cutch, leaves, bark and other cellulosic materials, adulterants and other impurities, such as sand and earth or dirt.

3.1.1 It shall be dried and pressed in the form of cubes, or irregular blocks or thick flakes of light rust-brown to dark-brown in colour, and fluffy in mass.

3.1.2 It shall be astringent, of a slightly bitter taste and free from unpleasant odours. When examined under microscope, a freshly-broken surface shall exhibit numerous shining and acicular crystals of catechin.

3.2 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods given in col 6 and 7.

4. PACKING AND MARKING

4.1 The material shall be suitably packed in gunny bags or in wooden cases as agreed between the purchaser and the supplier.

4.2 The containers shall be legibly and indelibly marked with the following:

a) Name of the material and its grade;

b) Name of the manufacturer or his registered trade-mark, if any; and

c) Net weight.

4.2.1 The material may also be marked with the Standard Mark.

4.2.2 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

TABLE 1 REQUIREMENTS FOR *KATTHA*

(*Clauses* 2.1 *and* 3.2)

Sl No.	Characteristic	Requirement for			Methods of Test, Ref to	
		Grade 1	Grade 2	Grade 3	Cl No. in IS : 2962-1964*	Appendix A
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Loss on drying, percent by weight, *Max*	12	12	12	6	—
ii)	Catechin content, percent by weight, *Min*	55	40	30	7	—
iii)	Matter insoluble in rectified spirit, percent by weight, *Max*	25	25	25	9	—
iv)	Insolubles in boiling water, percent by weight, *Max*	3	6	7·5	11	—
v)	Total ash, percent by weight, *Max*	1·3	3·5	3·5	12	—
vi)	Acid insoluble ash, percent by weight, *Max*	0·2	0·5	0·5	13	—
vii)	Poisonous metals:					
	a) Arsenic, ppm, *Max*	1	1	1	—	A-2
	b) Lead, ppm, *Max*	2·5	2·5	2·5	—	A-3
	c) Copper, ppm, *Max*	30	30	30	—	A-4
	d) Zinc, ppm, *Max*	50	50	50	—	A-5
	e) Tin, ppm, *Max*	250	250	250	—	A-6

*Methods of sampling and test for *KATTHA*.

5. SAMPLING

5.1 The method for drawing representative samples and for preparation of samples for test shall be as given in 2 of IS : 2962-1964*.

5.2 Number of Tests

5.2.1 Tests for catechin content shall be conducted on each of the individual samples.

5.2.2 Tests for all the other requirements shall be conducted on the composite sample.

*Methods of sampling and test for *KATTHA*.

5.3 Criteria for Conformity

5.3.1 *For Individual Sample* — The lot shall be declared to have satisfied the requirements for the catechin content tested on the individual samples if $\bar{x} - 0{\cdot}6\ R \geqslant$ the specified value for each declared grade.

where

$$\bar{x} = \frac{\text{the sum of individual test results}}{\text{number of test results}}, \text{ and}$$

R = the difference between the maximum and the minimum test results.

5.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics, the test results on the composite sample shall have to meet the corresponding specified requirements.

APPENDIX A

(*Table* 1)

METHODS OF TEST FOR POISONOUS METALS IN *KATTHA*

A-1. QUALITY OF REAGENTS

A-1.1 Pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect test results.

A-2. DETERMINATION OF ARSENIC

A-2.1 Preparation of Test Solution — Weigh accurately 10 to 20 g of the composite sample (*see* **2.3.3** of IS : 2962-1964†), transfer to a 500-ml Kjeldahl digestion flask and wet with 10 to 15 ml of cencentrated nitric acid. Add 5 to 10 ml of concentrated sulphuric acid and heat cautiously. Add nitric acid dropwise from the pipette to hasten the oxidation of the material. When the oxidation is complete and the solution is colourless, add 20 ml of water and boil till the solution strongly fumes. Cool and dilute with water to 50 ml in a graduated flask. Use a suitable aliquot (not exceeding 30 ml) estimated to contain 0·1 to 0·3 mg of arsenic, for the determination of arsenic in accordance with the method prescribed in IS : 2088-1962‡. Preserve the rest of the solution for the determination of other metallic impurities.

*Specification for water, distilled quality (*revised*).

†Methods of sampling and test for *KATTHA*.

‡Modified Gutzeit method of test for arsenic.

A-2.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length of the stain as well as intensity of its colour is not greater than those produced with a standard arsenic solution containing 0·1 mg of arsenic (as As).

A-3. DETERMINATION OF LEAD

A-3.0 General — Two methods for estimation of lead are prescribed, both based on the dithizone method, of which any one may be used; but in case of dispute, the photometric method described in **A-3.1** shall be used.

A-3.1 Photometric Method

A-3.1.0 *Outline of the Method* — The material is subjected to rapid wet oxidation for complete destruction of organic matter, and then earthy phosphates are precipitated and iron prevented from interfering by the addition of ammonium citrate and by rendering the solution alkaline with ammonia. Interference by other metals, is prevented by the addition of potassium cyanide and the lead is extracted by chloroform and dithizone. Excess reagent is removed from the chloroform by alkaline cyanide. The optical density of the chloroform layer is then read photometrically. Finally, lead is estimated by interpolation on the standard curve obtained by plotting the results with known quantities of lead.

A-3.1.1 *Reagents*

A-3.1.1.0 All reagents, including water, should be lead-free.

A-3.1.1.1 *Concentrated nitric acid* — sp gr 1·42, conforming to IS : 264-1950*.

A-3.1.1.2 *Concentrated sulphuric acid* — sp gr 1·84, conforming to IS : 266-1961†.

A-3.1.1.3 *Ammonium citrate solution* — 25 percent, solution in water (*w/v*).

A-3.1.1.4 *Sodium hexametaphosphate solution* — 10 percent solution in water (*w/v*).

A-3.1.1.5 *Thymol blue indicator solution* — 0·04 percent (*w/v*). Warm 0·1 g of thymol blue with 4·3 ml of 0·05 N sodium hydroxide and 5 ml of 90 percent ethanol; when dissolution is complete, add sufficient 20 percent ethanol to produce 250 ml of solution.

A-3.1.1.6 *Ammonium hydroxide solution* — sp gr 0·880.

A-3.1.1.7 *Potassium cyanide solution* — 10 percent solution in water (*w/v*). This solution should be at least 2-day old, so that traces of sulphide may become oxidised.

*Specification for nitric acid.

†Specification for sulphuric acid (*revised*).

A-3.1.1.8 *Hydroxylamine hydrochloride solution* — 20 percent solution in water (*w/v*).

A-3.1.1.9 *Chloroform* — Shake 250 ml of chloroform with 25 ml of water containing 1 ml of 10 percent (*w/v*) potassium cyanide solution and about 20 drops of 5 N ammonium hydroxide. Separate and reject the aqueous layer, wash the chloroform with water, and filter.

A-3.1.1.10 *Dithizone stock solution* — 0·1 percent (*w/v*) solution of diphenyl-thiocarbazone (dithizone) in chloroform. Filter and store in a refrigerator.

A-3.1.1.11 *Dilute dithizone solution* — Shake 6 ml of the dithizone stock solution with 9 ml of water and 1 ml of 5 N ammonium hydroxide. Separate and reject the lower layer and spin the aqueous layer in a centrifuge until clear. Prepare this solution freshly on the day of use.

A-3.1.1.12 *Nitric acid* — Dilute 1 volume of nitric acid (sp gr 1·42) diluted to 100 volumes with water.

A-3.1.1.13 *Ammoniacal sulphite-cyanide solution* — Mix 340 ml of ammonium hydroxide, 75 ml of 2 percent (*w/v*) sodium sulphite (Na_2SO_3) solution, 30 ml of potassium cyanide solution and 605 ml of water. The strength of these reagents are critical.

A-3.1.1.14 *Standard stock solution of lead* — Dissolve 1·60 g of pure lead nitrate in water, add 10 ml of concentrated nitric acid, and dilute to 1 litre.

A-3.1.1.15 *Standard dilute solution of lead* — Dilute 1 volume of the stock solution of lead to 100 volumes with water. Prepare the dilute solution freshly as required. One millilitre of this solution is equivalent to 0·01 mg of lead.

A-3.1.2 *Apparatus*

A-3.1.2.1 *Photoelectric absorption-meter* — fitted with filters having transmission at 520 mμ, or as given in **A-3.1.2.2**.

A-3.1.2.2 *Spectrophotometer* — instrument adjusted for measuring transmission at 520 mμ.

A-3.1.2.3 *Kjeldahl digestion flask* — 500-ml capacity.

A-3.1.3 *Procedure*

A-3.1.3.1 Take a suitable aliquot of the solution prepared in **A-2.1**, add 5 ml of ammonium citrate solution and 10 ml of sodium hexametaphosphate solution. Add a few drops of thymol blue indicator solution and sufficient ammonium hydroxide solution to give the blue-green colour indicating *p*H 9·0 to 9·5. Cool, add 1 ml of potassium cyanide solution and, if much iron is present, add 1 ml of hydroxylamine hydrochloride solution. Transfer the solution to a 100-ml separating funnel containing 10 ml of chloroform and rinse with a few millilitres of water. The volume of the aqueous layer at this stage should be approximately 50 ml. Add 0·5 ml of dilute dithizone solution, shake vigorously for 1 minute, and allow to

separate. If the lower layer is red, add dilute dithizone solution, until after shaking, a purple, blue or green colour is obtained. Run the chloroform layer into a second separating funnel, and wash through with 1 or 2 ml of chloroform. Add to the liquid in the first separating funnel 3 ml of chloroform and 0·2 ml of dilute dithizone solution. Shake vigorously for 30 seconds, allow the chloroform layer to separate, and add it to the main chloroform extract. This last chloroform extract should be green. If it is not, further extractions with chloroform and dithizone must be made until the green colour of the final extract indicates that all the lead has been extracted. Reject the aqueous layer. Add 10 ml of dilute nitric acid to the combined chloroform extracts, and shake vigorously for 1 minute. Allow to separate and reject the chloroform layer as completely as possible. To the nitric acid layer left in the separating funnel add 30 ml of ammoniacal sulphite cyanide solution, exactly 10 ml of chloroform and 0·5 ml of dilute dithizone solution. Shake vigorously for 1 minute and allow to settle. Run off a little of the chloroform layer. Insert a plug of cotton-wool into the dry stem of the funnel and, after rejecting the first runnings, fill a 1-cm spectrophotometer cell with the chloroform solution.

Measure the optical densities of the test and blank solutions against chloroform (all in 1-cm cells) with a photoelectric absorption meter fitted with filters that possess a maximum transmission at or near 520 mμ with a band width of 23 mμ at 50 percent transmission or with a spectrophotometer at 520 mμ. Read the number of micrograms of lead equivalent to the observed optical densities of the test and blank solutions from previously prepared calibration graph, and so obtain the net amount of the lead in the sample.

A-3.1.3.2 Carry out a blank test simultaneously with all the reagents in the same proportions as in the text.

A-3.1.3.3 The calibration graph may be prepared as follows:

Measure 1·0, 2·0, 3·0, and 4·0 ml of standard dilute solution of lead into separating funnels and dilute each to 10 ml with dilute nitric acid. Proceed as described before. Measure the optical densities with chloroform in the comparison cell. Construct a graph relating the optical densities to the number of micrograms of lead.

A-3.2 Dithizone Method

A-3.2.1 *Reagents*

A-3.2.1.0 All reagents shall be free from lead.

A-3.2.1.1 *Citric acid* — solid.

A-3.2.1.2 *Dilute hydrochloric acid* — prepared by mixing equal volumes of water and concentrated hydrochloric acid (sp gr 1·16).

A-3.2.1.3 *Ammonium hydroxide solution* — sp gr 0·90.

A-3.2.1.4 *Potassium cyanide solution* — 10 percent (*w/v*), aqueous.

A-3.2.1.5 *Dithizone* (*diphenyl thiocarbazone*) *solution* — 0·1 percent (*w/v*) in chloroform.

A-3.2.1.6 *Concentrated sulphuric acid* — sp gr 1·84 (conforming to IS : 266-1961*).

A-3.2.1.7 *Concentrated nitric acid* — sp gr 1·42 (conforming to IS : 264-1950†).

A-3.2.1.8 *Absolute alcohol*

A-3.2.1.9 *Ethyl alcohol-water-sulphuric acid mixture* — in the proportion 10 : 20 : 1 (*v/v*).

A-3.2.1.10 *Ammonium acetate solution* — 10 percent (*w/v*), aqueous.

A-3.2.1.11 *Sodium sulphide solution* — 10 percent (*w/v*), aqueous, freshly prepared.

A-3.2.1.12 *Standard stock solution of lead* — Dissolve 0·16 g of pure lead nitrate in 50 ml of water, add 1 ml of concentrated nitric acid and make up to 100 ml with water. Reserve this solution in a well-stoppered bottle.

A-3.2.1.13 *Standard dilute solution of lead* — Dilute 2 ml of the standard stock solution of lead (*see* **A-3.2.1.12**) to 100 ml with water. This solution contains 0·02 mg of lead per millilitre. Prepare this solution fresh before use.

A-3.2.2 *Procedure*

A-3.2.2.1 *Separation of interfering metals* — Take a suitable aliquot of the prepared solution (*see* **A-2.1**), add 2 g of citric acid, heat just to boil and cool. Filter the solution through a Whatman No. 44 filter paper or its equivalent and wash the residue with small portions of hot dilute hydrochloric acid and finally with hot water. The volume of the filtrate should be kept as low as possible. Cool the solution in cold water, neutralize carefully to litmus with ammonium hydroxide solution and add 0·5 ml of ammonia in excess. Transfer the alkaline solution to a separating funnel, add 2 ml of potassium cyanide solution and extract immediately with 10, 10 and 5 ml portions of the dithizone solution, shaking vigorously for about 1 minute for each extraction. Separate the dithizone layer and wash the combined dithizone extracts once with about 10 ml of water. The dithizone solution contains all the lead.

A-3.2.2.2 *Conversion of the lead-dithizone complex to lead sulphate* — Evaporate off the dithizone extract in a hard-glass boiling tube over a water-bath. When all the chloroform has evaporated off, dissolve the residue in 0·7 ml of concentrated sulphuric acid and a few drops of concentrated nitric acid and heat cautiously to oxidize the organic matter. Add 1 to 2 ml of water and again heat to fuming. After oxidation, the solution containing

*Specification for sulphuric acid (*revised*).

†Specification for nitric acid.

the lead sulphate should be clear. Cool, add 5 ml of absolute alcohol and 10 ml of water and allow to stand for 18 hours.

A-3.2.2.3 Filter the lead sulphate (visible or not) on Whatman No. 44 filter paper or its equivalent washed with hydrochloric acid. Wash the filter paper twice with a few millilitres of ethyl alcohol-water-sulphuric acid mixture. Reject the filtrate and washings. Boil 10 to 15 ml of ammonium acetate solution in the tube in which the sulphate precipitation was carried out and pass the hot ammonium acetate solution through the filter to dissolve the lead sulphate. Pass and re-pass the same hot ammonium acetate solution through the filter paper several times to dissolve all the lead sulphate and wash the filter paper three times with 5-ml portions of hot water containing a little ammonium acetate. Transfer the combined filtrate to a Nessler cylinder having a mark at 50 ml. Into a similar Nessler cylinder (control) take 10 to 15 ml of ammonium acetate solution. To each of the Nessler cylinders, add 1·5 ml of ammonium hydroxide solution, 1 ml of potassium cyanide solution and make up the volume to 50 ml with water. Add 5 drops of sodium sulphide solution and mix. Add the standard dilute solution of lead (*see* **A-3.2.1.13**) from a burette to the control until the tints in the two Nessler cylinders match exactly. Note the volume of the standard lead solution added. Prepare another control tube containing all the reagents and also the volume of lead solution required in matching, dilute to the 50-ml mark with water and then add the sodium sulphide solution. Match the tint with that of the test solution.

A-3.2.2.4 Carry out a blank test simultaneously with all the reagents in the same proportion as for the test.

A-3.2.3 *Calculation* — From the volume of the standard lead solution used, calculate the lead content of the material as parts per million.

A-4. DETERMINATION OF COPPER

A-4.0 General — Of the two methods any one may be used but in case of dispute the photometric method described in **A-4.1** shall be used.

A-4.1 Photometric Method

A-4.1.1 *Apparatus*

A-4.1.1.1 *Spectrophotometer*

A-4.1.2 *Reagents*

A-4.1.2.1 *Citric acid* — solid.

A-4.1.2.2 *Ammonium hydroxide solution* — sp gr 0·880.

A-4.1.2.3 *Sodium diethyldithiocarbamate solution* — 0·1 percent (*w/v*), aqueous.

A-4.1.2.4 *Carbon tetrachloride* — redistilled.

A-4.1.2.5 *Sodium sulphate* — anhydrous.

A-4.1.2.6 *Dilute nitric acid* — concentrated nitric acid (*see* IS : 264-1950*) of sp gr 1·42 diluted with an equal volume of water.

A-4.1.2.7 *Standard copper solution* — Weigh accurately 0·100 0 g of pure copper turnings, carefully dissolve in the minimum amount of nitric acid, cool and dilute to 1 litre in a graduated flask. Pipette 10 ml of this solution into a 100-ml graduated flask and dilute to the mark. This solution contains 10 micrograms of copper per millilitre.

A-4.1.3 *Procedure*

A-4.1.3.1 Transfer a 10-ml aliquot of the test solution (*see* **A-2.1**) to a separating funnel. Add 1 g of nitric acid and dissolve it by shaking. Make the solution alkaline to litmus by adding ammonium hydroxide solution in small quantities. Add 5 ml of the sodium diethyldithiocarbamate solution, shake thoroughly and extract with 5-ml portions of carbon tetrachloride until the final extract is colourless. Dry the combined extracts by shaking thoroughly with anhydrous sodium sulphate. Filter the dry extract and wash the filter paper with carbon tetrachloride. Make up the volume of the filtrate to 25 ml with carbon tetrachloride and measure the absorption at 437 μm by means of the spectrophotometer.

A-4.1.3.2 Simultaneously, carry out blank determinations on the water and the reagents.

A-4.1.3.3 Prepare a series of standards by treating aliquots of the standard copper solution in the same manner as the test solution. From the absorption of the standard solutions, prepare a standard curve plotting absorption values against concentrations. From the curve, obtain the weight of copper present in the test solution.

A-4.2 Dithizone Method

A-4.2.1 *Reagents*

A-4.2.1.1 *Dilute hydrochloric acid* — prepared by mixing equal volume of water and concentrated hydrochloric acid.

A-4.2.1.2 *Ammonium hydroxide solution* — sp gr 0·880.

A-4.2.1.3 *Citric acid* — solid.

A-4.2.1.4 *Dithizone* (*diphenylthiocarbazone*) *solution* — 0·1 percent (*w/v*) in chloroform.

A-4.2.1.5 *Sodium diethyldithiocarbamate solution* — 0·2 percent (*w/v*) solution in water.

A-4.2.1.6 *Standard stock solution of copper* — Dissolve 0·392 5 g of pure crystallized copper sulphate in water ($CuSO_4.5H_2O$) and make up to 100 ml in a graudated flask; 1 ml contains 1 mg of copper (as Cu).

*Specification for nitric acid.

A-4.2.1.7 *Standard dilute solution of copper* — Dilute, when required, 1 ml of the above stock solution to 100 ml in a graduated flask. 1 ml contains 0·01 mg of copper (as Cu).

A-4.2.1.8 *Gum Arabic* — 1 percent (*w/v*) in distilled water, conforming to IS : 266-1961*.

A-4.2.1.9 *Concentrated sulphuric acid* — sp gr 1·84, conforming to IS : 264-1950†.

A-4.2.1.10 *Concentrated nitric acid* — sp gr 1·42.

A-4.2.2 *Procedure*

A-4.2.2.1 *Preparation of solution* — Weigh accurately 10 to 20 g of sample into a 500-ml Kjeldahl flask, add 5 to 10 ml of concentrated sulphuric acid followed by 5 to 10 ml of concentrated nitric acid and slowly heat the mixture. Add small amounts of nitric acid from time to time and continue heating until all organic matter is destroyed without charring. Remove nitric acid as far as possible by adding about 20 ml of water and reheating to fuming stage. Dilute the acid liquid with water, add 2 g of citric acid, neutralize exactly with ammonia (using a piece of litmus paper) and reacidify with 1 ml of concentrated hydrochloric acid. Cool and transfer to a separating funnel (total volume of the solvent should be about 100 ml).

A-4.2.2.2 *Extraction with dithizone* — Extract the copper by shaking with three successive portions of 5 ml of 0·1 percent solution of dithizone in chloroform, shaking thoroughly for a minute for each extraction. Separate the dithizone layer and wash the combined dithizone extracts with about 10 ml of water. Transfer the dithizone extract into a suitable neutral glass test tube and evaporate off the chloroform on water bath.

A-4.2.2.3 *Colorimetric determination of copper* — Heat the copper-dithizone residue in a test tube with 1 ml of concentrated sulphuric acid and a little concentrated nitric acid until all organic matter is destroyed. Add 5 ml of water and reheat to fuming stage. Cool, dilute with water and transfer the whole of the solution or a measured volume according to the amount of copper present, into a Nessler cylinder.

Add 1 ml of 5 percent citric acid solution, 4 ml of ammonium hydroxide solution followed by 5 ml of 1 percent gum Arabic solution and make up to 50 ml with water. Add 5 ml of a freshly made aqueous solution of sodium diethyl dithiocarbamate and match the colour by adding standard copper solution (containing 0·01 mg of Cu) to a control cylinder containing similar quantities of reagents as present in test solution.

A-4.2.2.4 Calculate the copper content of the sample from the known volume of the standard copper solution required for matching.

*Specification for sulphuric acid (*revised*).

†Specification for nitric acid.

A-5. DETERMINATION OF ZINC

A-5.0 General — Of the two methods any one may be used; but in case of dispute spectrophotometric method described in **A-5.1** shall be used.

A-5.1 Spectrophotometric Method

A-5.1.1 *Apparatus*

A-5.1.1.1 *Spectrophotometer*

A-5.1.2 *Reagents*

A-5.1.2.1 *Methyl red indicator solution* — 1 percent (*w/v*) aqueous.

A-5.1.2.2 *Copper sulphate solution* — Dissolve 8 g of copper sulphate ($CuSO_4.5\ H_2O$) in water and dilute to 1 litre. 1 ml of this solution contains 2 mg of copper.

A-5.1.2.3 *Ammonium hydroxide solution* — redistilled, sp gr 0·90.

A-5.1.2.4 *Concentrated hydrochloric acid* — sp gr 1·16.

A-5.1.2.5 *Hydrogen sulphide gas* — passed through a wash-bottle containing water.

A-5.1.2.6 *Dilute hydrochloric acid* — containing 5 percent (*w/v*) of concentrated hydrochloric acid.

A-5.1.2.7 *Bromine water* — saturated.

A-5.1.2.8 *Phenol red indicator solution* — prepared by dissolving 0·1 g of phenol red in 100 ml of rectified spirit (*see* 323-1959*).

A-5.1.2.9 *Hydrochloric acid* (1 : 1) — Dilute concentrated hydrochloric acid with an equal volume of water.

A-5.1.2.10 *Ammonium citrate solution* — Dissolve 225 g of ammonium citrate in water, make alkaline to phenol red with ammonium hydroxide and add 75 ml in excess. Dilute to 2 litres. Extract this solution immediately before use as follows:

> Add to the solution a slight excess of dithizone solution and extract with carbon tetrachloride until the solvent layer is clear bright-green. Remove the excess of dithizone by repeated extraction with chloroform and finally extract once more with carbon tetrachloride. (It is essential that excess dithizone be entirely removed, otherwise zinc will be lost during the elimination of cobalt and nickel.)

A-5.1.2.11 *Dimethylglyoxime solution* — Dissolve 2 g of dimethylglyoxime in 10 ml of ammonium hydroxide solution and 200 to 300 ml of water, filter and dilute to 1 litre.

A-5.1.2.12 *α-nitroso β-naphthol solution* — Dissolve 0·25 g of α-nitroso β-naphthol in chloroform and dilute to 500 ml with chloroform.

*Specification for rectified spirit (*revised*).

A-5.1.2.13 *Chloroform* — redistilled.

A-5.1.2.14 *Dithizone (diphenylthiocarazone) solution* — Dissolve 0·05 g of dithizone in 2 ml of ammonium hy droxide solution and 100 ml of water and extract repeatedly with carbon tetrachloride until the solvent layer is clear bright-green in colour. Discard the solvent layer and filter the aqueous portion through a washed ashless filter paper. (This solution is best prepared as needed since it is only moderately stable even when kept in the dark and under refrigeration.)

A-5.1.2.15 *Carbon tetrachloride* — redistilled.

A-5.1.2.16 *Hydrochloric acid* — approximately 0·04 N.

A-5.1.2.17 *Stock solution of zinc* — Dissolve exactly 0·500 g of pure dilute granulated zinc in slight excess of dilute hydrochloric acid and dilute to one litre in a graduated flask.

A-5.1.2.18 *Standard solution of zinc* — At the time of the experiment, dilute 10 ml of the stock solution of zinc (**A-5.1.2.17**) to 1 000 ml with hydrochloric acid (0·04 N). This solution contains 5 micrograms of zinc per millilitre.

A-5.1.3 *Procedure*

A-5.1.3.1 *Separation of sulphide group* — Take a suitable aliquot of the test solution (**A-2.1**) to contain 25 to 100 μ g of zinc, add 2 drops of methyl red indicator solution, 1 ml of copper sulphate solution and neutralize with ammonium hydroxide solution. Add sufficient quantity of concentrated hydrochloric acid to make the solution about 0·15 N with respect to this acid (about 0·75 ml excess in 50 ml of solution is satisfactory). The *p*H of the solution at this point should be 1·9 to 2·1 when measured with a glass electrode. Pass a steam of hydrogen sulphide gas into the solution until precipitation is complete. Filter the contents of the flask through a fine textured filter paper (Whatman No. 42 or equivalent) that has been previously fitted into the funnel and washed first with dilute hydrochloric acid and then with water. Collect the filtrate in a beaker and wash the flask and the filter with 3 to 4 small portions of water. Boil the filtrate gently until the odour of hydrogen sulphide is no longer detected; then add 5 ml of bromine water and continue boiling until bromine has been expelled. Allow the solution to cool, neutralize with ammonium hydroxide solution using phenol red as indicator and then make it slightly acidic with hydrochloric acid (1 : 1) by adding an excess of 0·2 ml. Dilute the resultant solution in a graduated flask to contain 0·2 ml to 1·0 μg/ml of zinc (as Zn).

A-5.1.3.2 *Elimination of nickel and cobalt* — Transfer a 20-ml aliquot of the solution to a separating funnel. Add to it 5 ml of ammonium citrate solution, 2 ml of dimethylglyoxime solution and 10 ml of α-nitroso β-naphthol solution and shake the contents of the funnel for 2 minutes. Discard the lower layer and extract the aqueous layer with 10 ml of chloroform to remove residual α-nitroso β-naphthol. Discard the chloroform layer.

A-5.1.3.3 *Isolation and estimation of zinc* — To the aqueous layer obtained after eliminating nickel and cobalt which at this point has a *p*H of 8·0 to 8·2, add 2·0 ml of the dithizone solution and 10 ml of carbon tetrachloride, and shake for 2 minutes. Allow the layers to separate and remove the aqueous layer as completely as possible, withdrawing it by means of a pipette attached to the vacuum line. Wash down the sides of the separating funnel with about 25 ml of water and draw off the aqueous layer without shaking. Add 25 ml of hydrochloric acid (0·04 N) to the carbon tetrachloride layer in the separating funnel and shake for 1 minute to transfer the zinc to the acid-aqueous layer. Drain off and discard the carbon tetrachloride layer taking care to dislodge and remove the drop that usually floats on the surface. To the acid aqueous layer, add 5·0 ml of ammonium citrate solution and 10·0 ml of carbon tetrachloride (*p*H of the solution at this point is 8·8 to 9·0). Add the requisite volume of the dithizone solution calculated as follows:

> Pipette 4·0 ml of the standard solution of zinc into a separating funnel, add to it 21 ml of hydrochloric acid (0·04 N) from a burette, 5 ml of ammonium citrate solution, 10·0 ml of carbon tetrachloride and then add the dithizone solution in 0·1 ml increments, shaking briefly after each addition until a faint yellow colour in the aqueous layer indicates a bare excess of the reagent. Note the total volume of the dithizone solution added. Multiply this volume by 1·5.

After the addition of dithizone solution, shake the separating funnel for 2 minutes. Pipette 5·0 ml of the carbon tetrachloride layer and transfer it to the spectrophotometer cell. Dilute the solution with 10·0 ml of carbon tetrachloride, mix and determine its transmission at 540 mμ (*see* Note). Pipette into a series of separating funnels 0, 1, 2, 3 and 4 ml of the standard solution of zinc and add the necessary volume of hydrochloric acid (0·04 N) to make 25 ml. Add into each separator 5·0 ml of the ammonium citrate solution and the calculated volume of the dithizone solution. Shake the separating funnel, pipette out 5·0 ml of the carbon tetrachloride layer and transfer it to the spectrophotometer cell. Dilute the solution with 10·0 ml of carbon tetrachloride, mix and determine its transmission at 540 mμ. Proceed in the same manner with the solutions contained in other separating funnels.

NOTE — Dilution may be made in a clean dry test-tube, if the design of the cell does not permit direct mixing.

A-5.1.3.4 Plot the transmittance for each of the series of separating funnels on logarithmic scale against concentration of zinc in micrograms present in 25 ml of the diluted standard zinc solution in the particular separating funnel and draw a smooth curve through the points. (Intercept of this curve may vary from day to day depending on the actual concentration of dithizone used in the final extraction, but the slope of the curve should remain essentially the same.) From this curve, obtain the weight of zinc in micrograms.

A-5.2 Dithizone Method

A-5.2.1 *Reagents* — All reagents including water should be zinc free.

A-5.2.1.1 *Concentrated sulphuric acid* — sp gr 1·84.

A-5.2.1.2 *Concentrated hydrochloric acid* — sp gr 1·16.

A-5.2.1.3 *Citric acid*

A-5.2.1.4 *Ammonium hydroxide* — sp gr 0·880.

A-5.2.1.5 *Dithizone (diphenylthiocarbazone) solution* — 0·1 percent (*w/v*) in chloroform.

A-5.2.1.6 *Chloroform*

A-5.2.1.7 *Standard zinc solution* — Dissolve 0·124 5 g of zinc oxide in a mixture of 10 ml of water and 1 ml of sulphuric acid, and dilute with water to 1 000 ml. 1 ml of this solution contains 0·01 mg of zinc (as Zn).

A-5.2.1.8 *Sodium diethylthiocarbamate solution* — 0·2 percent (*w/v*) in water.

A-5.2.2 *Preparation of Solution* — Weigh accurately about 5·0 g of sample (*see* **2.3.3** of IS : 2962-1964*) and oxidise in a round bottom flask, using 5 ml of sulphuric acid and the minimum quantity of nitric acid. Expel the residual nitric acid as far as possible by diluting with about 20 ml of water and reheating until free from sulphuric acid fumes. Dilute the solution again with about 20 ml of water and separate any residue either by centrifuging or filtration, extract with hot diluted hydrochloric acid (1 : 1) and wash with hot water. Add the acid extracts and washings to the main filtrate. Add 2 g of citric acid in the liquid and add ammonium hydroxide until acid is neutralized (test with a piece of litmus paper) and add 0·5 ml of ammonia in excess and transfer to a 250-ml separating funnel and adjust the total volume with washings to 100 ml.

A-5.2.3 *Extraction with Dithizone* — Extract the solution with 0·1 percent solution of dithizone using 10 ml for the first and 5 ml each for the second and third extractions (lead, copper and zinc are extracted). Wash the dithizone extracts in turn with about 10 ml of water in a smaller separator and collect the combined dithizone extracts in a third separator.

A-5.2.4 *Separation of Copper* — Separate lead and zinc from copper by extracting the dithizone solution twice with dilute hydrochloric acid, using a mixture of 20 ml of water and 2 ml of hydrochloric acid (1 N) for each extraction. Remove the traces of residual dithizone from the acid liquor by shaking with chloroform. Boil the acid extracts and washings to remove chloroform, and make upto 50 ml. Carry out a control or blank determination throughout with similar apparatus and quantities of reagents only.

A-5.2.5 *Procedure* — Transfer so much of the acid solution as will contain about 0·01 to 0·02 mg of zinc (as Zn) to a 100-ml pear-shaped separator and

*Methods of sampling and test for *KATTHA*.

if necessary, dilute to 20 ml. Introduce into a second separator a similar quantity of control solution. Add to each sufficient ammonia (1 N) solution to neutralize the free acid and leave an excess of 2 ml of N ammonia solution. Add 2·5 ml of sodium diethyldithiocarbamate solution to each. Add a weak solution of dithizone in chloroform (2 ml of approximately 0·1 percent solution diluted shortly before use to 50 ml with chloroform) to the test solution with shaking until the red colour first produced gives place to a purple violet. Add a similar volume of the weak dithizone solution to the control. Shake both separators well for about 15 to 30 seconds. Then add standard zinc solution gradually until, on shaking, the colours in the two separators match exactly.

NOTE — Great care must be taken to avoid errors due to traces of zinc derived from reagents and glassware. The control test should be carried through from start to finish in a manner closely similar to that of the test.

A-5.2.6 Calculate the zinc content in millilitres of standard zinc solution added to match the colour with the material.

A-6. DETERMINATION OF TIN

A-6.0 General — Of the two methods any one may be used but in case of dispute the photometric method described in **A-6.1** shall be used.

A-6.1 Photometric Method

A-6.1.1 *Reagents*

A-6.1.1.1 *Fusion mixture* — 3 parts anhydrous sodium carbonate and 1 part potassium cyanide.

A-6.1.1.2 *Dilute hydrochloric acid* — Mix equal volumes of water and concentrated hydrochloric acid.

A-6.1.1.3 *Diethylammonium diethyldithiocarbamate solution* — 1 : 20 in chloroform.

A-6.1.1.4 *Chloroform*

A-6.1.1.5 *'Dithiol' reagent* — Dissolve 0·1 g of dithiol in 2·5 ml of 5N-sodium hydroxide solution. Add 0·5 ml of thioglycollic acid and dilute to 50 ml with water.

A-6.1.1.6 *'Lorol' solution* — 1 percent aqueous solution of sodium lauryl sulphate.

A-6.1.1.7 *Standard tin solution* — Dissolve 1 g of pure tin metal in 100 ml of 1 : 1 hydrochloric acid and dilute with the same concentration of acid to 1 litre. 1 ml contains 1 mg of tin (as Sn). Prepare more dilute solutions when required.

A-6.1.2 *Preparation of Solution* — Weigh accurately 5 to 10 g of sample (*see* **2.3.3** of IS : 2962-1964*) depending upon tin content into a small

*Methods of sampling and test for *KATTHA*.

porcelain crucible. Dry and char the sample on a hot-plate, heat to ash in a muffle furnace at 600°C. Add 1 g of fusion mixture and fuse with the ash by holding the crucible with nickel tongs over a Bunsen burner. Cool the crucible. Place it in a small beaker and cover the latter with a watch glass. Add 10 ml of water and run 10 ml of dilute hydrochloric acid (1 : 1) cautiously into the crucible (fume cupboard). Boil the contents of the beaker gently for 30 minutes. Cool and filter. Wash the beaker and crucible with water.

A-6.1.3 *Separation of Copper* — Transfer the solution to a small separating funnel and add 5 ml of diethylammonium diethyldithiocarbamate reagent. Shake and run off the chloroform layer, extract the aqueous layer with successive 1 ml portions of the reagent until the chloroform layer is finally colourless. Wash the aqueous layer with a few millilitres of chloroform. Dilute the aqueous solution with water to 50 ml in a volumeteric flask.

A-6.1.4 *Procedure* — To 10 ml of the aqueous solution add 0·5 ml of dilute hydrochloric acid (1 : 1) and transfer it to a 20 ml volumetric flask and add in the order given; 1 drop of thioglycollic acid, 2 ml of concentrated hydrochloric acid and 0·5 ml of ' lorol ' solution and 1 ml ' dithiol ' reagent with thorough mixing after each addition. Place the flask in a water-bath at 60°C for 10 minutes. Cool and dilute the contents to the mark. Measure the optical density at 545 mμ against a reagent blank.

A-6.1.5 Construct a titration curve with the aid of standard tin solution and calculate the tin content.

A-6.2 Stannous Oxide Method

A-6.2.1 *Reagents*

A-6.2.1.1 *Ammonium hydroxide solution* — sp gr 0·90.

A-6.2.1.2 *Concentrated hydrochloric acid* — sp gr 1·16.

A-6.2.1.3 *Dilute sulphuric acid* — 1 : 3 by volume.

A-6.2.1.4 *Hydrogen sulphide gas* — passed through a wash bottle containing water.

A-6.2.1.5 *Wash solution* — Mix 100 ml of saturated ammonium acetate solution with 50 ml of glacial acetic acid and 850 ml of water.

A-6.2.1.6 *Ammonium polysulphide solution* — Pass hydrogen sulphide gas through 200 ml of ammonium hydroxide solution contained in a bottle immersed in ice-cold water until no more gas is absorbed. Add 200 ml of the ammonium hydroxide solution and dilute with water to make 1 000 ml. Add 25 g of flowers of sulphur to this solution and keep for several hours to digest the sulphur and then filter. Use the filtrate.

A-6.2.1.7 *Dilute acetic acid* — Dilute 1 volume of glacial acetic acid with 9 volumes of water.

A-6.2.2 *Procedure*

A-6.2.2.1 Take a suitable aliquot of the test solution (**A-2.1**), dilute to 400 ml, cool and add ammonium hydroxide solution until the contents are alkaline. Add 20 ml of either concentrated hydrochloric acid or dilute sulphuric acid. Cover the beaker with a watch glass. Heat the solution to about 95°C and pass a slow stream of hydrogen sulphide gas through it for one hour. Digest the mixture at 95°C for 1 hour and allow it to stand for another 30 minutes. Filter the contents of the beaker through a quantitative filter paper and wash the residue of stannous sulphide on the filter, alternatively with three portions each of wash solution and hot water. Transfer the residue along with the filter paper to a 50-ml beaker, add 10 to 20 ml of the ammonium polysulphide solution, heat to boiling and filter by decantation. Add 10 ml of the ammonium polysulphide solution, boil and filter again. Repeat this once more and finally wash the filter paper with hot water. Acidify the combined filtrate and washings with dilute acetic acid, gently boil for 1 hour and allow to stand overnight. Filter the resulting mixture through a 11-cm ashless filter paper. Wash the filter paper alternately with two portions each of the wash solution and hot water. Transfer the residue along with the filter paper to a tared porcelain crucible and dry it thoroughly in an air-oven. Carefully ignite the filter paper, using a Bunsen flame and incinerate the contents to convert the sulphide to oxide. Partly cover the crucible and heat strongly over a Bunsen or Meker burner. The sulphide shall be gently roasted to the oxide which may then be heated to a high temperature without loss by volatilization.

A-6.2.2.2 Cool the crucible in a desiccator and weigh as stannous oxide (SnO_2). Repeat the process of heating, cooling and weighing till the difference between two successive weighings is less than 1 mg. Note the lowest weight. Obtain the weight of metallic tin from the weight of stannous oxide by using the factor 0·787 7.

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